AD NUMBER

ADB000729

NEW LIMITATION CHANGE

TO

Approved for public release, distribution unlimited

FROM

Distribution authorized to U.S. Gov't. agencies only; Test and Evaluation; 04 OCT 1974. Other requests shall be referred to Naval Surface Warfare Center, White Oak, Code 242, Silver Spring, MD 20910.

AUTHORITY

usnswc ltr, 16 jul 1979

NOLTR 74-177

NOLTR 74-177

NOL TECHNICAL REPORT

A CHARACTERIZATION STUDY OF SEVERAL HEAT RESISTANT EXPLOSIVES

BY
E.E. Kilmer

4 October 1974

NAVAL ORDNANCE LABORATORY
WHITE OAK, SILVER SPRING, MARYLAND 20910

NAVAL ORDNANCE LABORATORY
WHITE OAK, SILVER SPRING, MARYLAND 20910

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER NOLTR 74-177	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE *(and Subtitle)* A CHARACTERIZATION STUDY OF SEVERAL HEAT RESISTANT EXPLOSIVES		5. TYPE OF REPORT & PERIOD COVERED Final - 1 Jul 1973-30 Jun 74 6. PERFORMING ORG. REPORT NUMBER NOLTR 74-177
7. AUTHOR(s) E. EUGENE KILMER		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Surface Weapons Center White Oak, Silver Spring, Maryland 20910		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NOL-0841/NASA-T-8917E
11. CONTROLLING OFFICE NAME AND ADDRESS		12. REPORT DATE 4 October 1974 13. NUMBER OF PAGES V + 29
14. MONITORING AGENCY NAME & ADDRESS*(if different from Controlling Office)*		15. SECURITY CLASS. *(of this report)* UNCLASSIFIED 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Distribution limited to U.S. Government Agencies only; test and evaluation. Other requests for this publication must be referred to ~~NOL, Code 244~~ NSWC, Code 242

HOLT 74

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

New Explosives
Thermally Stable Explosives
Explosive Properties

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Three heat resistant explosives were chosen as candidates for study of their physical and chemical characteristics. As a part of this study, the chemical synthesis and detonation properties were investigated to determine their suitability in applications to small explosive components. These secondary explosive candidates were subjected to temperatures approaching 315°C (600°F). The best candidate was selected for further study.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE
S/N 0102-014-6601

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

NOLTR 74-177 4 October 1974

A Characterization Study of Several Heat Resistant Explosives

This is a report on "A Characterization Study of Several Heat Resistant Explosives" work being conducted for the Lyndon B. Johnson Spacecraft Center at Houston, Texas, under Task NOL-0841/NASA-T-8917E, which covered the period 1 July 1973 - 30 June 1974.

This work is being carried out to characterize several new thermally stable explosives and to determine their usefulness in explosive components for possible application in the Space Shuttle Program. The latest findings on the thermal and explosive properties of these new materials are discussed.

The author wishes to acknowledge the explosive synthesis study by Dr. Thomas Hall and Mr. Michael Sitzmann, vapor pressure measurements by Mr. David Covell, chemical analysis by Dr. Donald Kubose, electrostatic sensitivity results by Mr. Louis Montesi, and vacuum thermal stability results by Mr. Herbert T. Simmons, Sr., of the Naval Ordnance Laboratory. The identification of commercial materials implies no criticism or endorsement by the Naval Ordnance Laboratory.

ROBERT WILLIAMSON II
Captain, USN
Commander

I. Kabik

I. KABIK
By direction

TABLE OF CONTENTS

TABLES

TABLE OF CONTENTS (Continued)

TABLES

ILLUSTRATIONS

1.0 BACKGROUND

1.1 This is the Annual Report for Fiscal 1974 on the "Space Shuttle Explosives" task being conducted for NASA, Lyndon B. Johnson Space Center at Houston, Texas. This work was carried out for NASA during the fiscal year under task NOL-0841/NASA. The work in part was accomplished in order to complete Phase I of the NASA work statement and to accelerate Phase II. The purpose of the work is to cover the development of a secondary explosive suitable for use in spacecraft hardware which is subjected to extremely high temperature and vacuum conditions. The design goal criterion shall be no significant degradation of the explosive after 600°F/1-hour exposure. It is anticipated that the total program will be accomplished over several phases of work. During the initial Phase I of the program several candidate explosives will be chemically and physically characterized. This characterization shall include as a minimum:

a. Melting Point Determination

b. Vacuum Thermal Stability

c. Shock Sensitivity

d. Electrostatic Sensitivity

e. Detonation Velocity

1.2 During Phase II the following is to be accomplished:

a. Completion of the characterization work on a selected candidate explosive,

b. Establish the compatibility of the explosive with materials to be defined,

c. Study explosive synthesis to insure feasibility of production and technical adequacy of the explosive product.

2.0 PROCUREMENT OF HEAT RESISTANT EXPLOSIVE CANDIDATES

The Naval Ordnance Laboratory (NOL) has begun to implement the above work statement in the following ways:

2.1 The selection of prime candidates which could possibly meet the high temperature requirement was followed by the partial characterization of three high explosive materials. Two of these materials were synthesized at NOL and the other was procured from the Los Alamos Scientific Laboratory (LASL).

2.2 The synthesis of two thermally stable explosives TPT (2,4,6-tripicryl-s-triazine) Figure 1 and ONT (2,2",4,4',4",6,6',6"-Octanitro-m-terphenyl) Figure 2 was accomplished at the NOL. The latest finding of a thermally stable explosive of interest is attributed to LASL. This material is named PYX (2,6-bis (Picrylamino)-3,5-dinitropyridine) Figure 3. According to LASL, this explosive has properties comparable to TPT. NOL procured between one and two pounds of this explosive for study. NOL had NASA concurrence (T. Graves, EP-4) on this matter.

2.3 The physical and chemical properties to be determined are:

a. Melting point range,

b. Bulk density,

c. Heat of combustion (refs. 4, 5),

d. Heat of vaporization (ref. 6),

e. Vacuum thermal stability (ref. 7),

f. Compatibility with metals and organic chemicals,

g. Chemical analysis according to typical specification WS5003, i.e., percent of surface moisture and volatile materials, percent of water insolubles, percent acetone insolubles (ref. 8).

2.4 The detonation properties will be determined by:

a. Impact Sensitivity (shock sensitivity)

b. Small Scale Detonation Velocity (ref. 9)

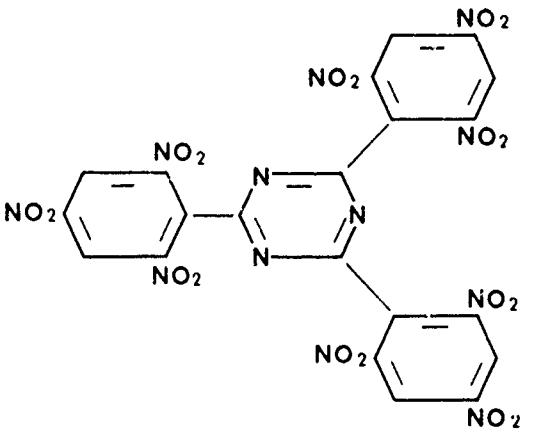
c. Small Scale Gap Test Sensitivity (various densities; ref. 10)

d. Electrostatic Sensitivity

e. Non-Reactive Hugoniot (ref. 11)

The data collected above should provide information for judgement of the candidate to best satisfy the design criterion of "no significant degradation of the explosive" and provide information for establishing a procurement specification.

CANDIDATE



2,4,6 TRIPICRYL-s-TRIAZINE

TPT

NOL

SYNTHESIS: REF 1

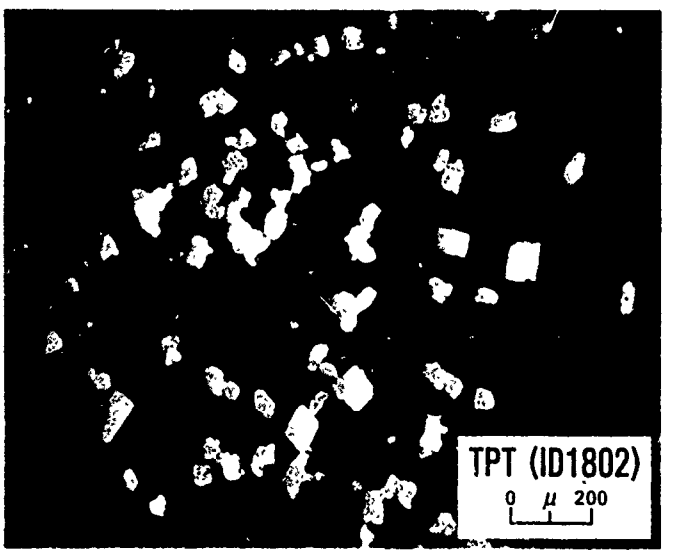
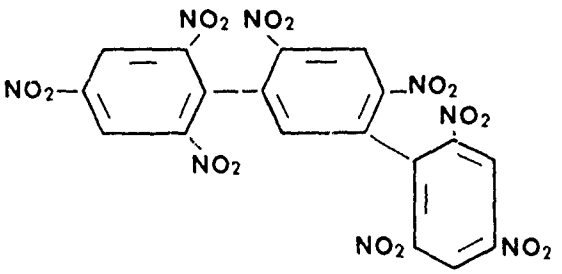


FIG. 1 CHEMICAL STRUCTURE AND PHOTOMICROGRAPH OF TPT

CANDIDATE



2,2'',4,4',4'',6,6',6''-OCTANITRO-m-TERPHENYL

ONT

NOL

SYNTHESIS: REF 2

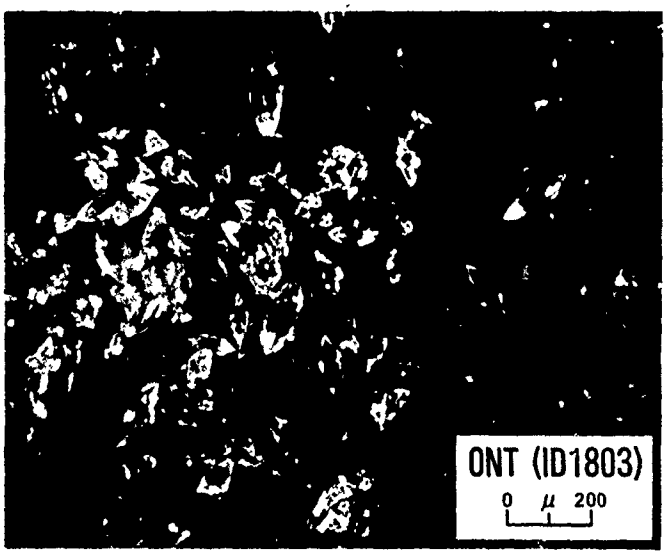
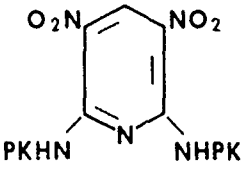


FIG. 2 CHEMICAL STRUCTURE AND PHOTOMICROGRAPH OF ONT

CANDIDATE



2,6-BIS (PICRYLAMINO)-3,5-DINITROPYRIDINE

PYX

LASL

SYNTHESIS: REF 3

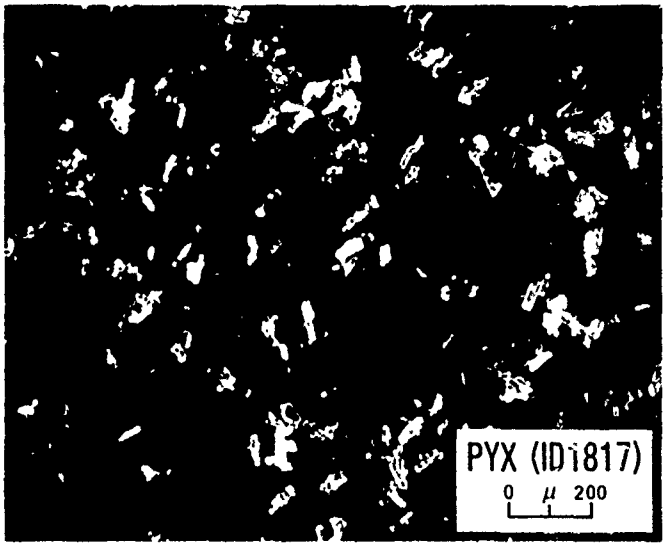


FIG. 3 CHEMICAL STRUCTURE AND PHOTOMICROGRAPH OF PYX

3.0 EXPLOSIVE SYNTHESIS STUDY

3.1 In an effort to accelerate the synthesis program, the manpower was concentrated toward the preparation of single lots of material which could be studied for chemical purity and handling characteristics. This, in part, means a study of each chemical synthesis to determine how realistic and practical it is to prepare a pound of explosive and apply these techniques to "scale-up" procedures.

3.2 A considerable amount of time was required to recrystallize ONT (octanitro-m-terphenyl) to insure that the material had very high purity. After many recrystallizations, the results from the vacuum thermal stability tests (Table 1) were higher than those of earlier preparations at NOL. A limited study was made to determine the cause. Gas chromatograph-Mass Spectrometer (GC-MS) studies indicated the presence of nitrobenzene (the crystallization solvent) in the amount of approximately 0.04%. This was due to occluded solvent which was not removed by drying at 150°C under vacuum because of the very high boiling point (210°C) of nitrobenzene. The larger the particle size, the greater the amount of occlusion. Previous samples were of uniformly smaller particle size because they were prepared in smaller quantities (smaller volumes of nitrobenzene) and were cooled more rapidly. It is very difficult to rapidly cool large volumes of nitrobenzene at temperatures of approximately 200°C with conventional laboratory equipment.

3.3 The sample of PYX, 2,6-bis (Picrylamino-3, 5-dinitropyridine), prepared by LASL showed poor vacuum thermal stability. After reviewing the results of Table 1, LASL was contacted to verify the published data on the material. We were informed that the "purity" of the sample was questionable because it had not been recrystallized and had been dried only at 70°C. In an effort to repurify the sample, some of it was recrystallized from 90% nitric acid and dried under vacuum for 16 hours at 150°C.

3.4 Vacuum Thermal Stability tests have been made on PYX which has been purified by recrystallization from 90% nitric acid. The results are shown in Table 1. TPT was also run at the same time for comparison. Under the conditions of this test, PYX began to show some decomposition at 300° (572°F) and definite decomposition at 315°C (600°F). By comparison TPT is thermally stable at 315°C.

4.0 VAPOR PRESSURE MEASUREMENTS OF TPT, ONT and PYX

4.1 A series of measurements has been completed, using a modified version of the apparatus described in references 6 and 15. The objective of these measurements was to determine vapor pressure as a function of temperature for TPT, ONT and PYX, and from the vapor pressure data, to calculate heats of sublimation for these explosives. A secondary objective was to obtain at least qualitative data in regard to the physical and chemical stability of these materials when exposed to temperatures up to 300°C.

TABLE 1 VACUUM THERMAL STABILITY TEST RESULTS

(SUMMARY)

EXPLOSIVE	TEMPERATURE °C	20 MINUTE SURGE (cc/g)	TWO HOUR PERIOD (cc/g/hr)
TPT	280	0.29	0.10
(NOL ID 1802)	300	0.37	0.29
	315	0.50	1.20
PYX	280	3.21	0.46*
(NOL ID 1817)	280	1.27	0.64**
	300	1.10	2.10***
	315	1.41	7.06***
ONT	280	0.28	0.63
(NOL ID 1803)	300	0.88	3.94

*Material as received from LASL.
**After drying 16 hrs @ 150° C.
***After recrystallization from 90% nitric acid and dried 16 hrs @ 150° C.

4.2 Samples were prepared by packing portions of the material to be tested into receptacles which had been machined into stainless steel sample trays. The receptacles were 0.5 in. in diameter by 3/32 in. deep. For the packing, a hydraulic ram was used at a pressure of 14,000 lbs. per sq. in. After weighing the samples, they were placed in a glass vacuum manifold and heated by passing vapors from a boiling, refluxed, organic solvent through a jacket around the vacuum manifold. A given sample was heated for a period of time, Δt, usually several hours in duration, under vacuum and then removed from the manifold and re-weighed. Vacuum, during the heating process was maintained in the range 2×10^{-7} torr to 2×10^{-6} torr.

4.3 Vapor pressures were computed from the observed weight loss. The expression used was;

$$P(\text{torr}) = 17.14\ G(T/M)^{1/2}$$

where G is the weight loss in grams/cm^2/sec.; T is the absolute temperature, and M is the molecular weight. The molar heat of sublimation, ΔH_s (kCal/mole), was calculated from the following relationship:

$$\log \frac{P_2}{P_1} = \frac{\Delta Hs}{4.576}\left(\frac{T_2 - T_1}{T_1\ T_2}\right)$$

where P_1, P_2, and T_1, T_2, represent the coordinates of two points on the curve relating vapor pressure and temperature. Table 2 shows the solvents and the corresponding temperatures used in this work. Benzyl Benzoate decomposed at its boiling point and could not be used for the experiment. A comparable material was not available.

4.4 Table 3 summarizes the data obtained by these measurements. All of the TPT samples withstood the heating very well, i.e., there was no marked change in the physical appearance of the samples as a result of the heating. All of the ONT samples, however, were broken up and granular in consistency; typically the upper layer of the sample was dislodged from the rest of the sample. No material appeared to be lost in the manifold by means other than evaporation. The break up of the sample caused large errors in the estimate of surface area shown in Table 2. The estimate of vapor pressure shown in Table 2 does not take into account this change in surface area. All of the PYX samples were badly broken up and material was physically strewn about the interior of the manifold. Under these conditions, no applicable determination of weight loss was possible, and no estimate of vapor pressure could be made. From direct observation, it appeared that this break-up phenomenon occurred during the first 15-20 minutes of heating.

4.5 TPT, PYX, and ONT after being subjected to the high temperatures were analyzed mass spectrographically, and the maximum decomposition that took place was estimated to be less than 2.5%. This figure quickly dropped to insignificance as lower temperatures were used.

TABLE 2 SOLVENTS AND CORRESPONDING TEMPERATURES USED IN VAPOR PRESSURE DETERMINATIONS

SOLVENT	BOILING POINT (°C)	SAMPLE TRAY TEMP (°C)
METHYL ACETATE	57.4	56.9
WATER	100.0	98.9
ANISOLE	152.7	149.7
NITROBENZENE	210.3	206.9
NONANOIC ACID	253.0	247.6
HEXADECANE	287.5	277.8
BENZYL BENZOATE*	323.0	-

*Not used because the solvent tends to decompose badly at the boiling temperature.

TABLE 3 SUMMARY OF DATA OBTAINED AND VAPOR PRESSURES CALCULATED IN THE VAPOR PRESSURE STUDY

SAMPLE	MOL. WT.	TEMP. (°C)	Δ t (sec)	SURFACE AREA (cm^2)	WT. LOSS (gm)	VAPOR PRESSURE (torr)
TPT	714	277.8	55,500	6.3	0.1268	$4.77(10^{-6})$
		247.6	157,380	5.1	0.0227	$4.26(10^{-7})$
		247.6	235,100	5.1	0.0303	$3.70(10^{-7})$
		206.9	239,400	5.1	0.0024	$2.73(10^{-8})$
		206.9	768,900	5.1	0.0041	$1.48(10^{-8})$
		98.9	149,040	5.1	INDETERMINATE	
ONT	590	277.8	38,400	5.1	0.1760	$1.50(10^{-5})$
		247.6	56,220	6.3	0.0189	$8.57(10^{-7})$
		247.6	55,200	5.1	0.0185	$1.07(10^{-6})$
		247.6	190,920	5.1	0.0341	$5.67(10^{-7})$
		206.9	71,040	5.1	0.0021	$9.01(10^{-8})$
		206.9	229,020	5.1	0.0041	$5.46(10^{-8})$
		149.7	492,900	5.1	INDETERMINATE	
PYX	621	277.8	56,100	5.1	*	
		206.9	56,400	5.1	*	
		149.7	63,720	5.1	*	

*Samples broken up and scattered around within the manifold.

4.6 Figures 4 and 5 show the vapor pressure versus temperature function for TPT and ONT respectively. The expected logarithmic function is clearly apparent in the TPT data, and from these data, the molar heat of sublimation was calculated to be 43.9 kCal/mole. The ONT data do not show the proper form, and this fact is believed to be due to sample break-up phenomenon described above which makes an estimate of surface area impossible. Since no meaningful surface area data were available, no attempt was made to calculate the heat of sublimation for ONT.

5.0 CHEMICAL ANALYSIS OF EXPLOSIVES AFTER PROLONGED HEAT TREATMENT UNDER VACUUM

5.1 In an attempt to measure the heats of sublimation of TPT, ONT and PYX, the explosives were heated at elevated temperatures under high vacuum (10^{-6} to 10^{-7}mm) in NOL's vapor pressure apparatus. To ascertain whether or not decomposition had occurred, chemical analysis of the explosive residue for each explosive was made.

5.2 Analyses of the residues were made by the ethylenediamine/dimethylsulfoxide spectrophotometric technique and compared to the starting materials as standards. In addition, the spectral envelope for each compound near the maximum absorption was measured and also compared to the standard.

5.3 The results obtained are as follows in Table 4. In all cases the spectral envelopes were the same as the standards.

6.0 DETONATION PROPERTIES

6.1 Detonation velocity measurement is an important tool in determining how an explosive will perform. For this task the explosive is being tested in column diameters of 0".1 and 0".2 in steel bodies 1".0 in O.D. The velocity is measured over a one inch length. The results of small scale detonation velocity measurements on PYX in the 0".1 column diameter are shown in Figure 6. Velocity measurements were made on ONT in 0".1 and 0".2 column diameters: on TPT in 0".1, 0".2, and 0".3 column diameters. The results are shown in Figures 7 and 8. All of these explosives appear to be performing as expected. The velocities are of the same magnitude as other heat-resistant explosives such as HNS. They are lower than RDX, for example, but this is to be expected since the detonation velocity and sensitivity are compromised for thermal resistance. Since these explosives are detonating in the 0".1 diameter column, it is conceivable that they will perform in detonating cord with a comparable diameter and very possibly at smaller diameters.

6.2 The shock sensitivity of TPT has been determined at various densities by the small scale gap test method. This test is an arbitrary configuration to study the shock sensitivity of small confined charges and thus the transfer of detonation between small-diameter confined charges. The results of the tests are shown in Table 5 (SSGT results are shown for ONT and PYX in Tables 6 and 7). By way of comparison, TPT has about the same shock sensitivity as HNS.

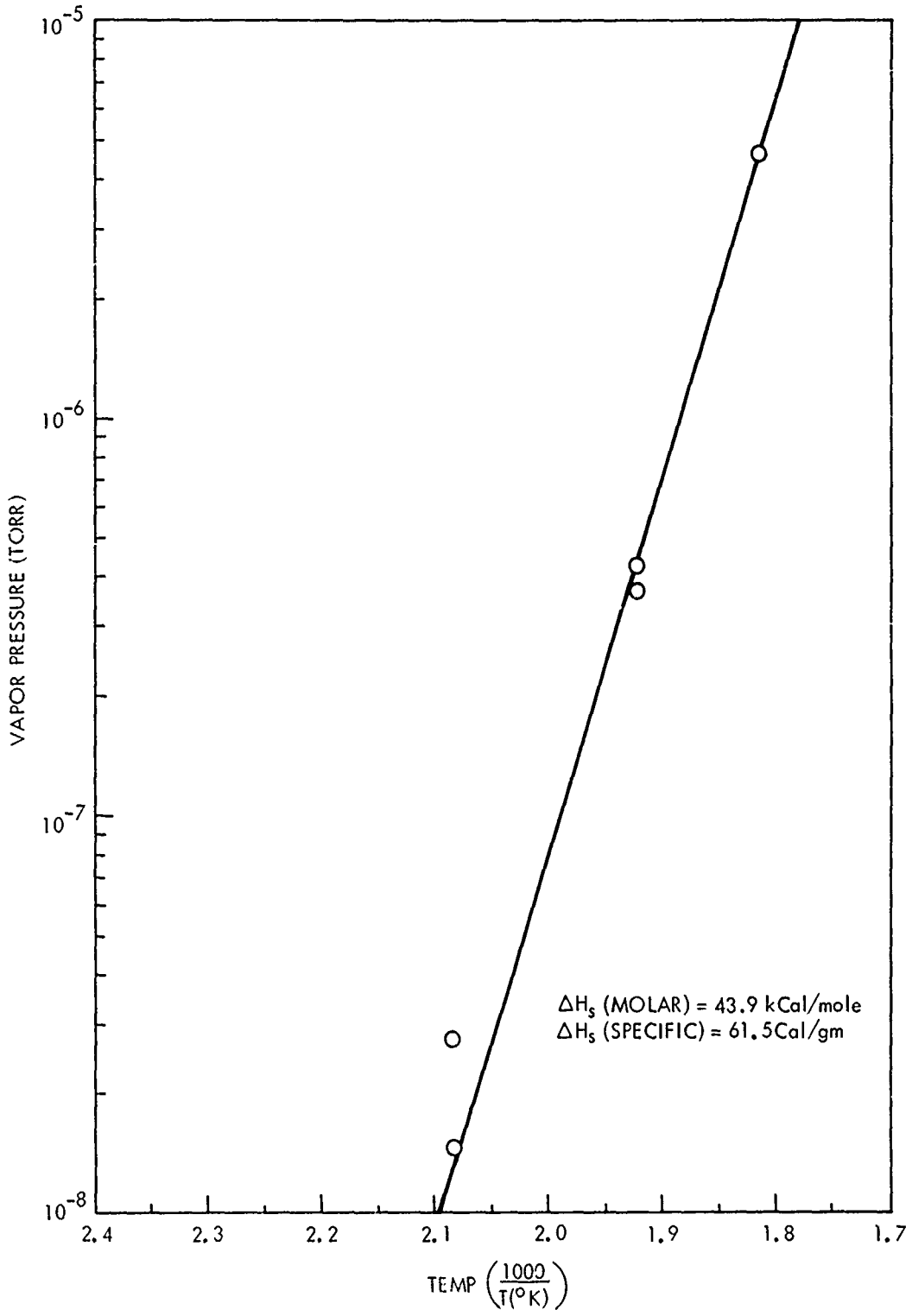


FIG. 4 VAPOR PRESSURE OF TPT

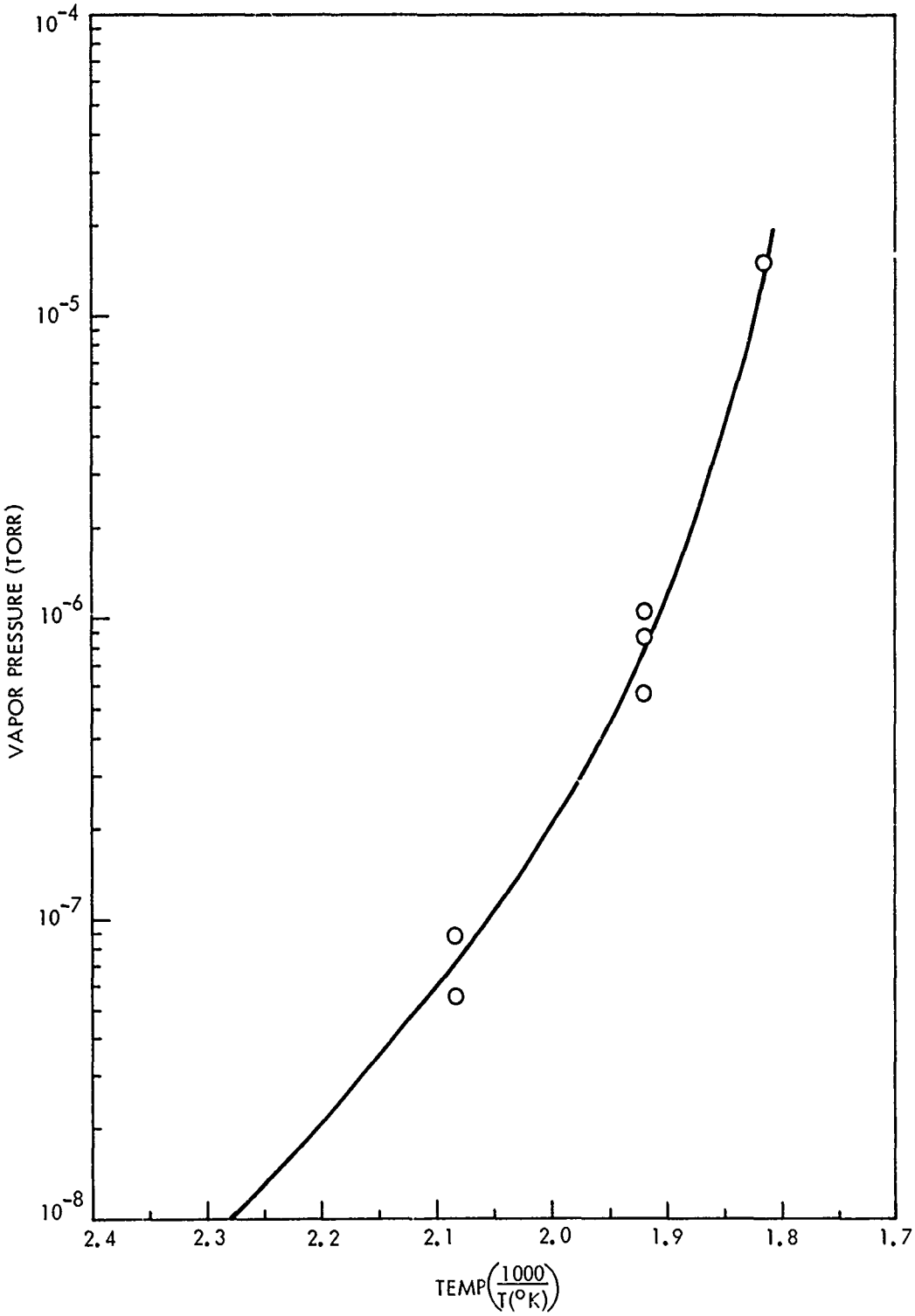


FIG. 5 VAPOR PRESSURE OF ONT

TABLE 4 ANALYSIS OF EXPLOSIVES AFTER HEAT TREATMENT UNDER VACUUM

EXPLOSIVE	TEMPERATURE, °C	TIME, HOURS	% EXPLOSIVE IN RESIDUE
TPT	278	16	97.1
ONT	278	16	96.7
PYX	278	16	96.8
TPT	250	44	99.3
ONT	250	44	98.7

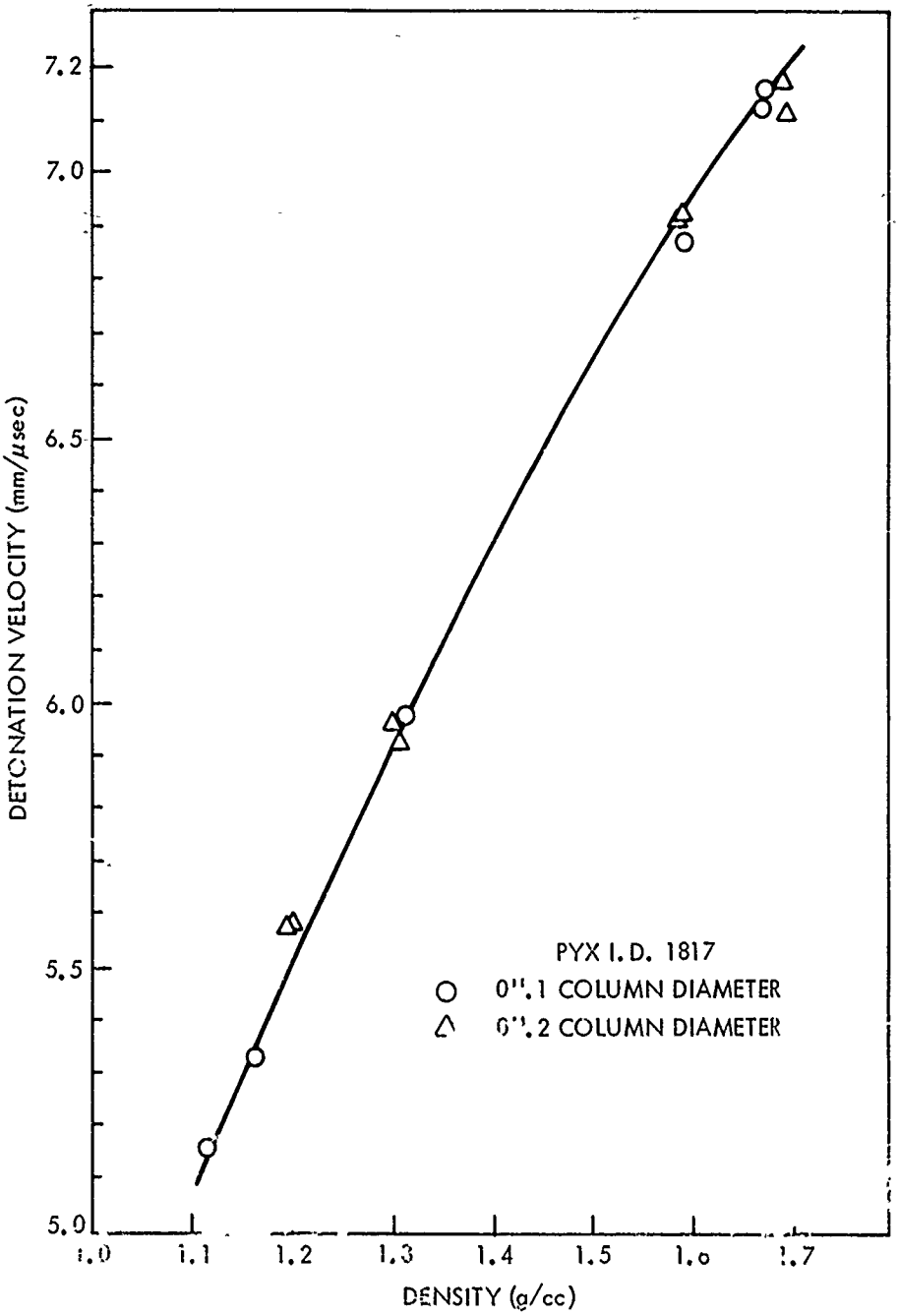


FIG. 6 THE SMALL SCALE DETONATION VELOCITY OF PYX AS A FUNCTION OF DENSITY AND COLUMN DIAMETER

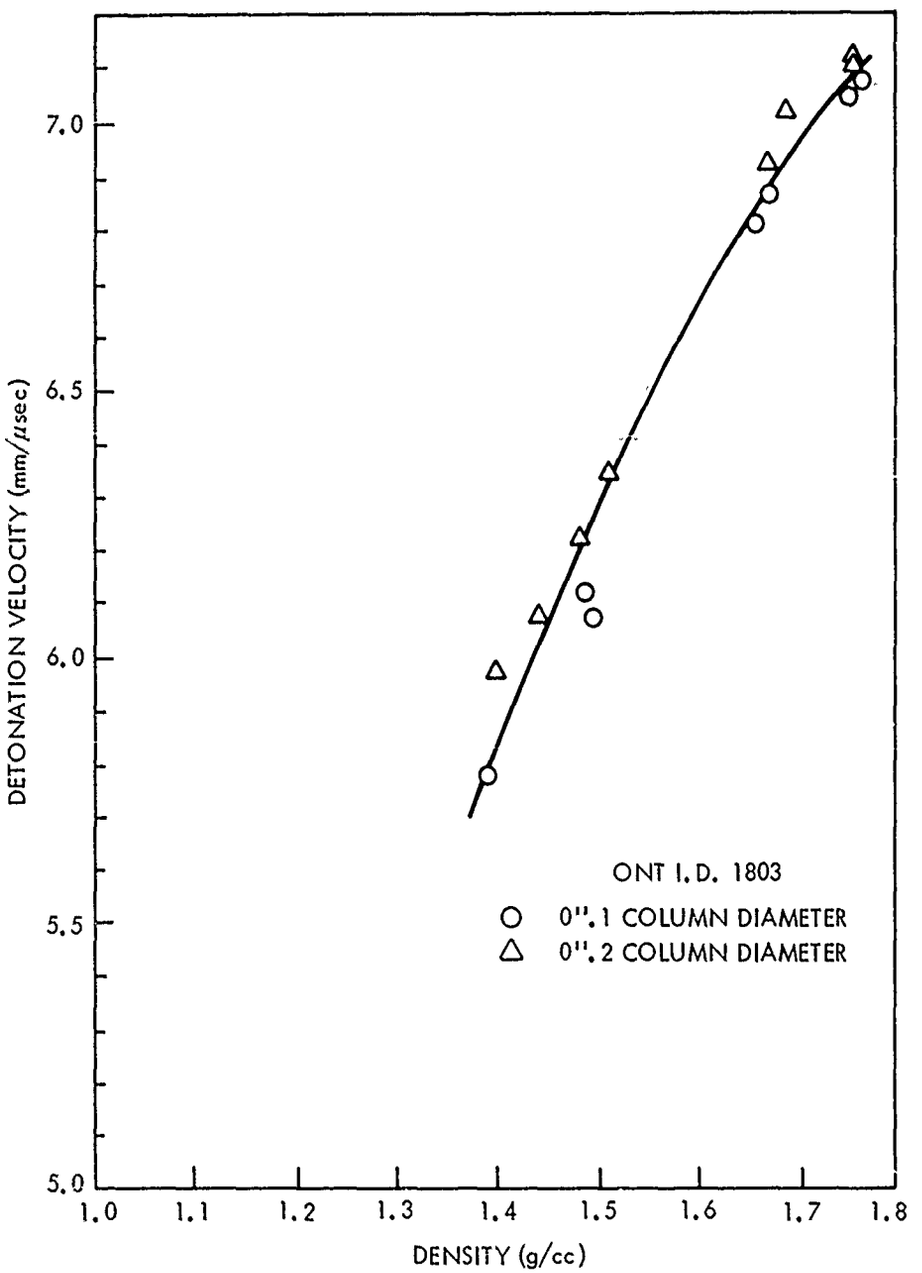


FIG. 7 THE SMALL SCALE DETONATION VELOCITY OF ONT AS A FUNCTION OF DENSITY AND COLUMN DIAMETER

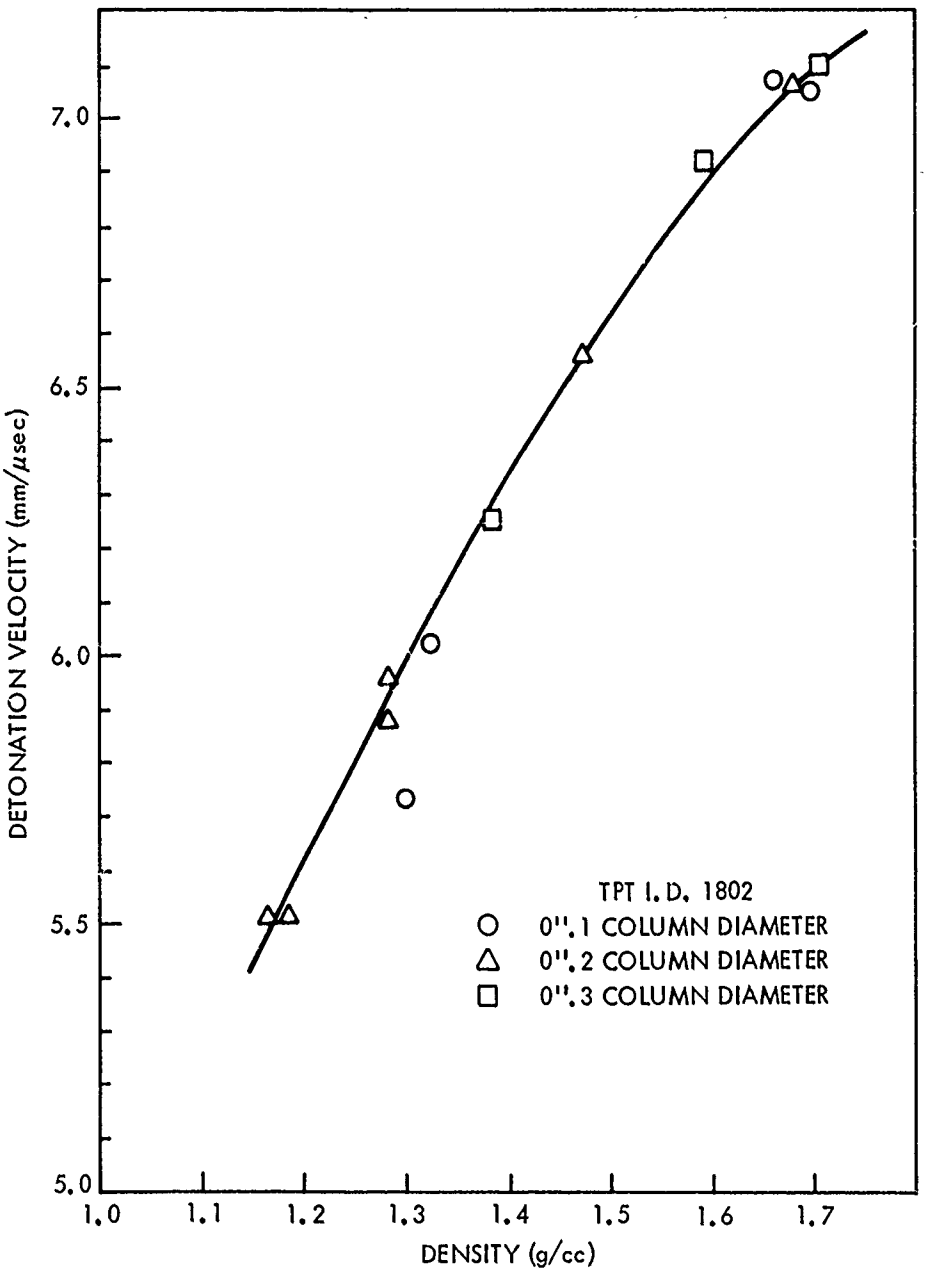


FIG. 8 THE SMALL SCALE DETONATION VELOCITY OF TPT AS A FUNCTION OF DENSITY AND COLUMN DIAMETER

EXPLOSIVE	TPT	L.O.	1417
TMD		I.D. NO.	1802

LOADING PRESSURE (KPSI)	DENSITY (GM/CM^3)		% TMD	SENSITIVITY (DBG) [1]			
	AVG.	s		AVG.	g	s_m	N
4	1.193	0.0557		4.800	0.0414	0.0281	20
8	1.242	0.0214		4.753	0.0312	0.0205	20
16	1.325	0.0496		5.059	0.0384	0.0257	20
32	1.444	0.0268		5.423	0.0798	0.0430	20
64	1.658	0.0111		7.217	0.0988	0.0530	20

[1] SENSITIVITY PARAMETERS ARE GIVEN IN REFERENCE 14

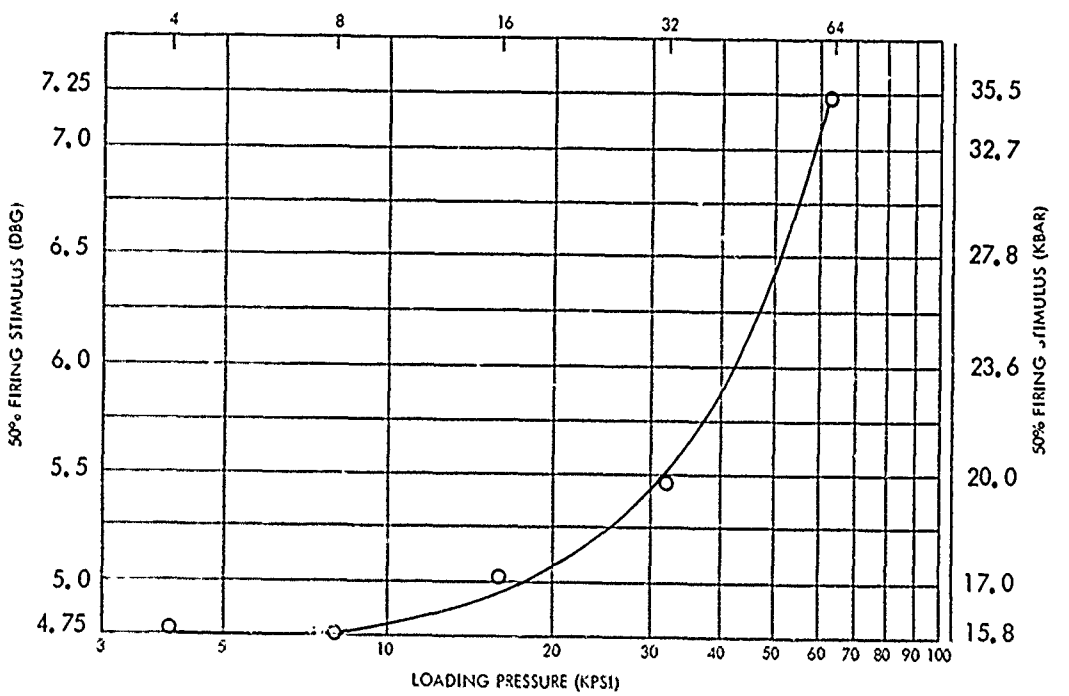


TABLE 5 SMALL SCALE GAP TEST (SSGT) DATA FOR TPT
(2, 4, 6 - TRIPICRYL 1-S - TRIAZINE)

EXPLOSIVE	ONT	L.O.NO.	1416
TMD		I.D.NO.	1803

LOADING PRESSURE (KPSI)	DENSITY (GM/CM^3)		% TMD	SENSITIVITY (DBG) [1]			
	AVG.	s		AVG.	g	s_m	N
4	1.396	0.0113		4.567	0.0617	0.03489	20
8	1.487	0.0082		4.752	0.0068	0.0070	20
16	1.595	0.0068		5.213	0.0071	0.0039	20
32	1.6775	0.0045		5.950	-	-	20
64	1.7688	0.0038		6.936	0.0276	0.0219	20

[1] SENSITIVITY PARAMETERS ARE GIVEN IN REFERENCE 14

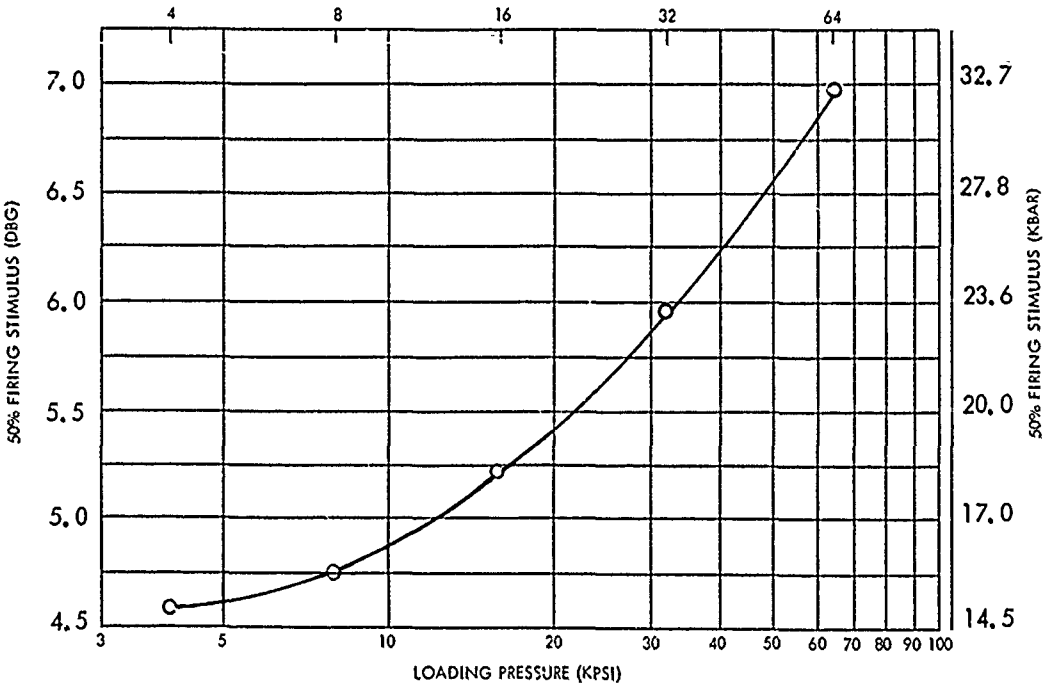


TABLE 6 SMALL SCALE GAP TEST (SSGT) DATA FOR ONT
(2, 2', 4, 4', 4", 6, 6', 6" - OCTANITRO - m - TERPHENYL)

EXPLOSIVE	PYX	L.O..	1402
TMD		I.D.NO.	1817

LOADING PRESSURE (KPSI)	DENSITY (GM/CM^3)		% TMD	SENSITIVITY (DBG)[1]			
	AVG.	s		AVG.	g	s_m	N
4	1.0788	0.0336		4.758	0.0502	0.0312	21
8	1.2298	0.0119		5.122	0.0251	0.0190	20
16	1.3691	0.0140		5.410	0.0567	0.0338	20
32	1.5154	0.0082		5.734	0.0498	0.0283	20
64	1.6754	0.0042		6.599	0.0268	0.0208	20

[1] SENSITIVITY PARAMETERS ARE GIVEN IN REFERENCE 14

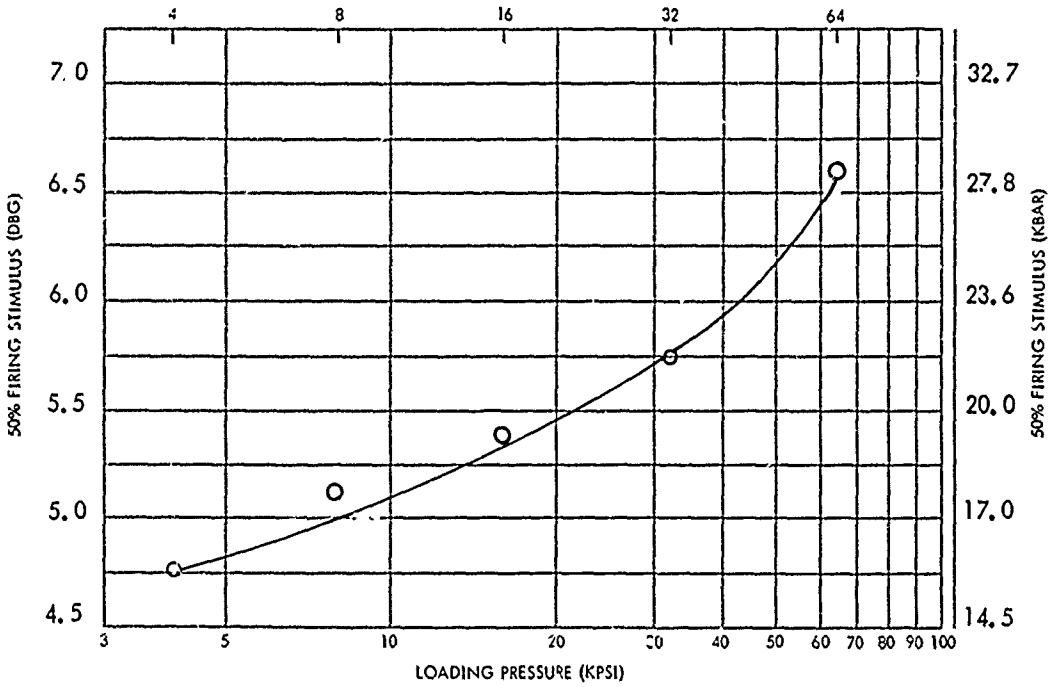


TABLE 7 SMALL SCALE GAP TEST (SSGT) DATA FOR PYX
(2, 6 - BIS (PICRYLAMINO) - 3, 5 - DINITROPYRIDINE)

6.3 The results of impact tests are often used as a guide to the handling sensitivity of explosives. The results of tests on TPT and PYX are shown in Table 8 along with the values for TNT and tetryl. The tests were made on standard Type 12 tools, sandpaper and 2.5 Kg weight. As can be seen, the heat resistant explosives are somewhat less sensitive than the common booster explosive tetryl but more sensitive than main charge explosives like TNT.

7.0 EXPLOSIVE/METAL AND EXPLOSIVE/ORGANIC MATERIAL COMPATIBILITY TESTS

7.1 The thermal stability of explosive materials at elevated temperatures is greatly affected by the materials in contact with them. Rosen and Simmons[12] have pointed out incompatibilities of high explosives with lead azide when heated to an elevated temperature range of 200-260°C. The decomposition was evidenced by evolution of gas and the formation of a black insoluble product which did not melt at 475°C. The reaction was interpreted as an oxidation of the azide ion by the nitro groups and must be considered as a limitation on the use of azides in intimate contact with typical organic explosives at elevated temperatures.

7.2 Since the explosives being considered will be used at temperatures exceeding 260°C, it is imperative to consider the materials that contact them at various temperatures to ascertain whether a compatibility problem would exist.

7.3 TPT, ONT, and PYX are being considered for first application in detonating cords, i.e., mild detonating cords and flexible linear shaped charges. The current materials used for the sheathing of these cords are silver, aluminum, and lead/antimony alloy. In view of this, explosive/metal mixtures 50/50 by weight were prepared and submitted to the NOL vacuum thermal stability/compatibility test. Results are shown in Table 9.

7.4 The approach used for the selection of the explosive/organic material contact was to reduce the number of combinations for test by considering possible applications. If the explosive was to be stored in a non-metallic container at temperatures approaching 600°F, then most plastics would be eliminated. Sealing materials such as epoxy resins would be incompatible at elevated temperatures (particularly those which are amine cured).

7.5 A high temperature polymer pioneered at the NOL is being evaluated in an effort to improve the performance of reinforced plastic heat shields for advanced reentry vehicles. This cast polymer is polyphenylquinoxaline (PPQ). It has a potential application as a potting compound and may be used in impact extrusions to fabricate containers. The potential of PPQ lies in its high thermal stability (800°F compared to 400°F for phenolics) and its increased toughness over brittle phenolics.

TABLE 8 IMPACT SENSITIVITY TEST RESULTS*

EXPLOSIVE	50% HEIGHT (cm)
ONT (NOL X-559) M. P. ABOVE 400°C	64
TPT (NOL ID 1802) M. P. 352°C	74
PYX (NOL ID 1817) M. P. 360°C	94
TETRYL**	40
TNT**	160

*Type 12 tools, sandpaper, 2.5 Kg wt.
**Established in earlier test runs.

7.6 The results of compatibility tests using a TPT/PPQ 90/10 mixture of powders are encouraging between 300°C and 315°C. The test results for the explosive/polymer mixtures are very good as shown in Table 9.

7.7 This study will be expanded to cover various stages of polymerization of the PPQ during which times excess solvent materials such as meta-cresol will be present and will be in contact with the explosive. This might be considered to be the environment for the explosive during a potting application.

8.0 ELECTROSTATIC SPARK SENSITIVITY OF EXPLOSIVE CANDIDATES

8.1 The electrostatic spark sensitivity test is one of the tests used to characterize and order the initiation sensitivity of explosives. Individuals and structural materials will accumulate static charges which when discharged might initiate sensitive explosives. The chance initiation by electrostatic spark discharge is therefore a real hazard in the handling and use of explosives.

8.2 TPT, ONT, and PYX were tested by two methods. The first method was in accordance with the procedures described in reference 13 The results are given in Table . The second method involved the use of the Wyatt apparatus, Four capacitors (1.0, 0.1, 0.01, and 0.001 mfd) were used with metal/metal (m/m) and metal/rubber (m/r) electrodes and test voltages of 7500, 5500, 4000, and 250 volts. The maximum no-fire test level was determined using a maximum of 20 trials at any one of the above capacitor-voltage test level. The criterion used for ignition was any evidence of smoke, flame, sparks, and/or burning of the explosive sample. The results of the test (Table 11) are indicative of explosives which are not sensitive to electrostatic spark discharge.

8.3 For comparison, a table of results from previous tests on primary and more sensitive high explosives is shown also in Table 11.

9.0 DETONATING CORDS

9.1 The first application of heat resistant explosives to be considered will be in small detonating cords. Detonation velocity results show that TPT, PYX, and ONT will perform in small diameters (0.2 explosive column) and hopefully will perform in low core load detonating cords. All three of the candidate explosives have been fabricated into 2-3 gr/ft. and 10-15 gr/ft. MDC using silver and aluminum sheaths. These cords will be subjected to elevated temperatures in subsequent tests.

TABLE 9 EXPLOSIVE PROPERTY MEASUREMENTS

(VACUUM THERMAL STABILITY AND COMPATIBILITY TESTS)

SAMPLE		STAB	COMP.	@ 260°C		@ 280°C		@ 300°C		@ 315°C	
				20 MIN GAS SURGE (cc/g)	2 hr. ml/g/hr	20 MIN GAS SURGE (cc/g)	2 hr. ml/g/hr	20 MIN GAS SURGE (cc/g)	2 hr. ml/g/hr	20 MIN GAS SURGE (cc/g)	2 hr. ml/g/hr
ONT (96-8151-82-2) ID 1803F		√				0.28	0.68	2.04	4.06		
PYX (AS REC.) ID 1817		√				3.21	0.46				
PYX (DRIED UNDER VACUUM 150°C)		√				1.27	0.64				
PYX (RECRY. 2X HNO_3)		√		0.44	0.29	0.59	0.51	1.06	2.09	1.41	7.06
TPT ID 1802		√				0.29	0.10	0.37	0.29	0.50	1.20
PPQ CONTROL		√				1.24	< 0.10	1.69	< 0.10		
TPT/PPQ 90/10			√			0.63	0.19	1.27	0.68	1.09	3.28
TPT/Ag (F-87196)	50/50		√			0.31	0.11	0.31	0.15	0.49	0.64
TPT/Al (#511-A)	50/50		√			0.23	0.12	0.34	0.20	0.51	0.62
TPT/Pb + Sb 94/6 #65680	50/50		√			0.19	0.10	0.26	0.14	0.31	0.55
TPT/STAINLESS STEEL (C-302)	50/50		√			0.31	0.16	0.34	0.33	0.47	0.78
ONT/Ag	50/50		√			0.22	0.38				
ONT/Al	50/50		√			0.25	0.35				
ONT/Pb + Sb	50/50		√			0.26	0.35				
ONT/STAINLESS STEEL	50/50		√			0.32	0.36				
PYX RECRY/Ag	50/50		√			0.39	0.19				
PYX RECRY/Al	50/50		√			0.35	0.16				
PYX RECRY/Pb + Sb	50/50		√			0.31	0.17				
PYX RECRY/STAINLESS STEEL	50/50		√			0.44	0.23				

TABLE 10 ELECTROSTATIC SENSITIVITY TEST RESULTS
(AS SPECIFIED BY NAVORD OD 44811)

TEST CONDITIONS:
POTENTIAL - 10K VOLTS
CAPACITANCE - 0.01 MFD

PASSED*	PYX	ID 1817
	TPT	ID 1802
	ONT	ID 1803

* OBSERVATION OF 20 CONSECUTIVE SHOTS WITH NO SMOKE, BURNING, AND/OR EXPLOSION.

TABLE 11 ELECTROSTATIC SPARK SENSITIVITY TEST RESULTS

CAP. (mfd)	HNS-TEFLON 7C 90/10 (ID 1494)		PYX (ID 1817)		TPT (ID 1802)		ONT (ID 1803)	
	m/m	m/r	m/m	m/r	m/m	m/r	m/m	m/r
1.0	2500	-	2000	-	4500	-	-	-
0.1	4000	4000	3000	2500	4500	4000	-	3000
0.01	5500	4500	6000	4000	7500*	6500	7500*	7500*
0.001	7500*	7500*	7500*	7500*	7500*	7500*	7500*	7500*

CAP. (mfd)	DEX. LEAD AZIDE		PETN		RDX	
	m/m	m/r	m/m	m/r	m/m	m/r
1.0	250	500	2000	3500	2000	4000
0.1	500	1500	2500	4500	5500	5500
0.01	1000	4000	7000	7500*	7000	7500*
0.001	2500	7500*	7500*	7500*	7500*	7500*

NOTE:

1. Unless otherwise specified, values given are maximum no fire values for Capacitance/Voltage level given (20 shot data)

*Maximum test voltage, no fires observed at this level.

10.0 CONCLUSIONS AND RECOMMENDATIONS

10.1 Considering the synthesis of both ONT and PYX, the results of the vacuum thermal stability tests are indicative of occluded solvents in the crystals and would result in poor performance of these materials at 300°C and above. TPT should be stable for 2 hours at 315°C, and longer.

10.2 Vapor pressure measurement was attempted on TPT, ONT, and PYX. PYX began to break up in the equipment, as did ONT, resulting in indeterminate values of surface area. At the limit of the test, (with temperature and pressure) TPT performed the best of the three candidates and would be expected to show little decomposition at 315°C.

10.3 TPT, ONT, and PYX perform satisfactorily in small column diameters of 0.2 and 0.1. These materials should perform well in small detonating cords of 3 - 15 gr/ft.

10.4 The results of the small scale gap test are indicative of relatively insensitive explosive performance and show that these materials are in the booster category for shock sensitivity. They have about the same sensitivity as Hexanitrostilbene (HNS).

10.5 All of these materials are safe to handle from the viewpoint of electrostatic sensitivity. They pass the Navy OD 44811 tests for acceptability in fuze applications and show no indication of burning in the Wyatt apparatus.

10.6 Compatibility at elevated temperatures is always of concern, but TPT, ONT, and PYX are compatible with silver, aluminum, lead alloy, and stainless steel at temperatures comparable to normal decomposition of the individual explosive. TPT is also compatible with a high temperature polymer, PPQ.

10.7 It can be concluded from the results of tests to date, that TPT is the most likely of the three explosive candidates to survive the high temperature requirement goal of 315°C (≃ 600°F) and perform satisfactorily after exposure for 2 hours.

10.8 However, it is recommended, that since the expected application will be in detonating cords, the screening of all three candidates continue. Detonating cords fabricated with these materials should be exposed to elevated temperatures to determine their thermal limitations of performance.

11.0 REFERENCES

1. Dacons, J. C., and Sitzmann, M. E., "Heat Resistant Explosives XXVI; The Synthesis and Properties of 2,4,6-Tripicryl-s-Triazine, TPT," NOLTR 68-64, 29 May 1968

2. Dacons, J. C., "Heat Resistant Explosives XXIII; The Preparation and Properties of 2,2",4,4',4",6,6',6"-Octanitro-m-Terphenyl, ONT," NOLTR 66-179, 5 Oct 1966

3. Coburn, M. D., and Singleton, S. L., "Picrylamino-Substituted Heterocycles .V. Pyridines (1,2)," J. Heterocyclic Chem., 9, 1039 (1972)

4. Barooday, E. E., Carpenter, G. A., Robb, R. A., and Zimmer, M. F., "Heats of Formation of 1,1,1-Trinitro-4,4-bisdifluoroaminopentane and 2-Fluoro-2,2-dinitroethanol by Combustion Calorimetry," J. Chem. Eng. Data, 13, 215 (1968)

5. Barooday, E. E., and Carpenter, G. A., IBID, 18, 28 (1973)

6. Rosen, J. M. and Dickinson, C., "Vapor Pressures and Heats of Sublimation of Some High Melting Organic Explosives," J. Chem. Eng. Data, 14, 1, 120 (Jan 1969)

7. Rosen, A. H., and Simmons, H. T., "Improved Apparatus and Techniques for the Measurement of the Vacuum Stability of Explosives of Elevated Temperatures," NAVORD Rpt 6629, 12 Mar 1959

8. WS5003, Purchase Description, HNS Explosive, Naval Ordnance Laboratory, White Oak, Silver Spring, Maryland

9. Hampton, L. D., and Stresau, R. H., "Small Scale Technique for Measurement of Detonation Velocities," NAVORD Rpt 2282, 31 Dec 1952

10. Ayres, J. N., "Standardization of the Small Scale Gap Test Used to Measure the Sensitivity of Explosives," NAVWEPS Rpt 7342, 16 Jan 1961

11. Pastine, D. J., and Bernecker, R. R., "P,v,E,T Equation of State for 1,3,5-Triamino-2,4,6-Trinitrobenzene," J. Appl. Phys., 45, 10 (Oct 1974)

12. Rosen, J. M. and Simmons, H. T., Jr., "Compatibility of Inorganic Azides with Organic Explosives," I. & EC Product Research and Development, 7, 262 (1968)

REFERENCES (Continued)

13. NAVORD OD 44811, "Safety and Performance Tests for Qualification of Explosives," 1 Jan 1972

14. Ayres, J., Montesi, L. J., Bauer, R. J., "Small Scale Gap Test (SSGT) Data Compilation: 1959-1972 Volume I Unclassified Explosives," NOLTR 73-132, 26 Oct 1973

15. Covell, D., "Vapor Pressures and Heats of Sublimation of Some High Melting Organic Explosives," NOLTR 74-136 (in progress)

DISTRIBUTION

Copies

Commander
Naval Sea Systems Command
Department of the Navy
Washington, D. C. 20360
SEA-0332
SEA-09G32

Commander
Naval Surface Weapons Center
Dahlgren Laboratory
Dahlgren, Virginia 22448
Technical Library

Director
Strategic Systems Projects Office
Department of the Navy
Washington, D. C. 20376
SSPO-2731 (M. Baron)

Commanding Officer
Naval Ordnance Station
Indian Head, Maryland 20640

Commander
Naval Weapons Center
China Lake, California 93555
Technical Library

Naval Academy
Annapolis, Maryland 21402
Hd. Weps Dept

Superintendent
Naval Postgraduate School
Monterey, California 93940

Commanding Officer
Naval Weapons Station
Yorktown, Virginia 23491
W. McBride
Library

Commanding Officer
Naval Ammunition Depot
Crane, Indiana 47522

DISTRIBUTION

	Copies
Commanding Officer Ballistics Research Laboratory Aberdeen Proving Ground Aberdeen, Maryland 21005 Technical Library	
Commanding General Army Materiel Command Department of the Army Washington, D. C. 20316	
Commander Army Rocket and Missile Agency Redstone Arsenal Huntsville, Alabama 35809	
Commanding Officer Frankford Arsenal Philadelphia, Pennsylvania 19137	
Director Defense Documentation Center Cameron Station Alexandria, Virginia 22314 TIPCR	12
Commander Systems Engineering Group RTD Wright-Patterson Air Force Base Ohio 45433	
Chief of Staff U.S. Air Force Washington, D. C. 20350 AFORD-AR	
NASA Lyndon B. Johnson Space Center Houston, Texas 77058	5

NOW-NOL - 5605/1 (REV. 6-74)

TO AID IN UPDATING THE DISTRIBUTION LIST
FOR NOL TECHNICAL REPORTS PLEASE COMPLETE
THE FORM BELOW

TO ALL HOLDERS OF NOLTR 74-177

by E. Eugene Kilmer, Code 242

DO NOT RETURN THIS FORM IF ALL INFORMATION IS CURRENT

A. FACILITY NAME AND ADDRESS *(OLD) (Show Zip Code)*

NEW ADDRESS *(Show Zip Code)*

B. ATTENTION LINE ADDRESSES:

C.

☐ REMOVE THIS FACILITY FROM THE DISTRIBUTION LIST FOR TECHNICAL REPORTS ON THIS SUBJECT.

D.

NUMBER OF COPIES DESIRED _____

GPO 875-354